



Interdiffusion Behavior of Aluminide Coated Two-Phase α_2 -Ti₃Al/ γ -TiAl Alloys at High Temperatures

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Abstract. Lower density materials of TiAl based intermetallic alloys have recently attracted intensive attention for the replacement of nickel-based superalloys used at high temperatures. As aluminium-rich titanium aluminide intermetallic compounds are normally brittle, two-phase α_2 -Ti₃Al/ γ -TiAl alloys have been developed. To increase the corrosion resistance of these alloy systems, an aluminide coating of TiAl₃ layer is normally applied. During operation at high temperatures, however, interdiffusion between the coating and the alloy substrate can occur and decrease the TiAl₃ layer thickness of the coating. The effects of temperature exposure on the growth of the TiAl₂ interdiffusion zone layer on two-phase α_2 -Ti₃Al/ γ -TiAl alloys with a chemical composition of Ti-47Al-2Nb-2Cr-0.5Y-0.5Zr are presented in this paper. The exponents for kinetics and rate constant of the TiAl₂ interdiffusion layer growth of this multi-component system were found under variation of temperature. The results were compared with those from other researchers.

Keywords: *coating degradation; interdiffusion; intermetallics; pack aluminide coatings; titanium-aluminide alloy.*

1 Introduction

Recently, intensive studies on titanium aluminide based intermetallic alloys have been conducted. These alloys were considered as replacements of nickel-based superalloys for application as turbine blades in aircraft gas turbine engines [1]. This group of materials has a lower relative density (3.9-4.1 gr/cm³) compared to nickel based superalloys [2]. In addition, these intermetallic materials have high creep and oxidation resistance [3], the two most important high-temperature material properties. However the fracture toughness of TiAl is relatively low. Therefore, in the last few decades increasing the toughness of two-phase α_2 -Ti₃Al/ γ -TiAl alloys has attracted attention to be further developed [4]. These alloys can be obtained by reducing the Al content in the TiAl alloys.

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As shown in the binary phase diagram of Ti-Al (Figure 1), the chemical composition of these alloys are in the range between 35 at% and 48 at% Al. Nevertheless, the capability of these alloys to form a protective layer of Al₂O₃ on the surface decreases when the aluminium content of the alloy is reduced. A solution for this problem has been proposed that uses a TiAl₃ based aluminide coating on a substrate of α_2 -Ti₃Al/ γ -TiAl alloys [5]. Coatings based on titanium aluminide TiAl₃ have been applied to high-temperature titanium based alloys, both as diffusion coating and overlay coating. The most extensively used diffusion-coating process is pack cementation as this process is relatively simple compared to the processes required to produce an overlay coating.

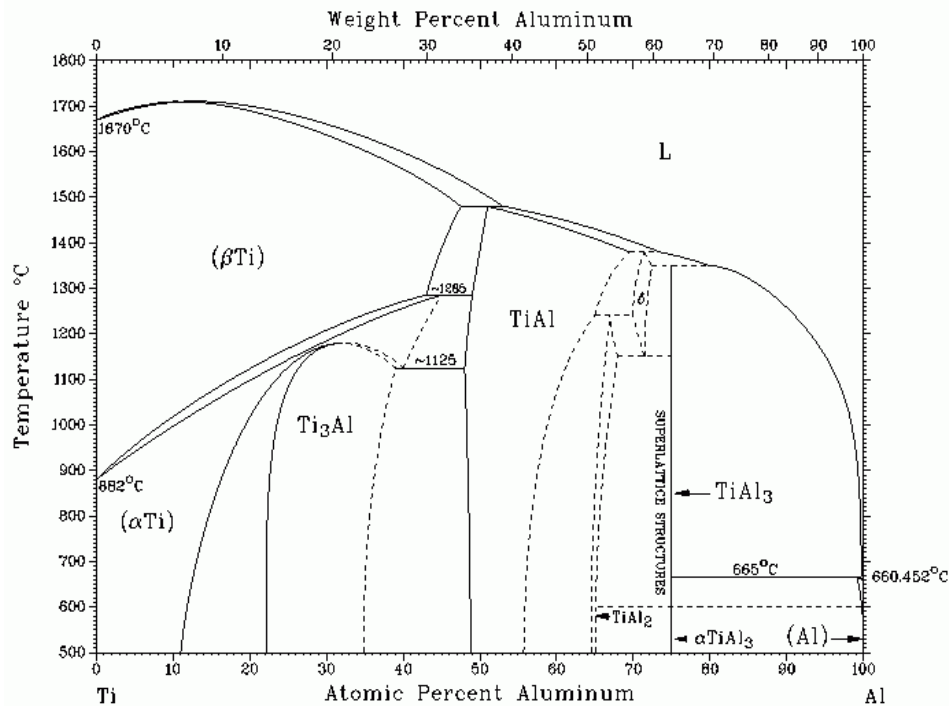


Figure 1 Equilibrium phase diagram of Ti-Al [6].

High activity pack aluminide coatings have been developed on a substrate of Ti-47Al-2Nb-2Cr-0.5Y-0.5Zr consisting of two-phase α_2 -Ti₃Al/ γ -TiAl [7]. At high-temperature operation, degradation of the coating system can occur through two different modes, i.e. high-temperature corrosion and interdiffusion of elements in the coated system. These two degradation modes reduce the aluminium content of the coating and consequently alter the ability of the coating to protect the substrate. Recently, oxidation at high temperatures as well

as hot corrosion mechanisms for coated α_2 -Ti₃Al/ γ -TiAl systems have been studied [5,7] and interdiffusion between an aluminide coating and α_2 -Ti₃Al/ γ -TiAl substrate has been modeled [8]. However, the mechanism of interdiffusion has not been well discussed. In normal applications, TiAl based alloys are subjected to temperatures of maximally about 900 °C. Nevertheless, during operation of an engine abnormal combustion or a malfunction could occur, leading to overheating and altering the performance of the coated materials. Several researchers [9-11] have studied interdiffusion in Ti-Al systems. However, they worked with relatively simple systems of binary Ti-Al. This study investigated the behavior of interdiffusion in the aluminide coated α_2 -Ti₃Al/ γ -TiAl system containing alloying elements of Cr, Nb, Zr and Y, at 800, 900 and 1000 °C.

2 Experimental Work

A two-phase α_2 -Ti₃Al/ γ -TiAl alloy was prepared by melting a mixture of pure elements in an argon-purged single arc furnace. The targeted alloy composition in atomic percent was Ti-47Al-2Cr-2Nb-0.5Zr-0.5Y. In order to obtain high homogeneity of the alloy, melting of the alloy button was done four times. Further homogenization was carried out by heating the as cast alloy button in a tube furnace at 1100 °C for 10 hrs. To avoid oxidation, the furnace was purged with high-purity argon gas. After quenching in water, the alloy button was cut into several sample coupons, each of which had 2.0 mm thickness. Prior to the pack cementation process, the surface samples were ground to a 2000 grit finish and then ultrasonically cleaned in acetone.

Pack cementation was used to develop coatings consisting of TiAl₃ on the surface of the substrate coupons. After a number of trials, the pack powder mixture consisted of three components, i.e. a master alloy of pure aluminium powder, an activator of NH₄Cl powder, and an inert filler of Al₂O₃ powder with a composition of 20 wt%, 2 wt%, and 78 wt%, respectively, was selected for coating production. Four coupon samples of the substrate were buried with the pack mixture in an alumina retort of 35 mm diameter and 70 mm length. The position of the coupon samples in the retort was arranged so that each sample was separated from the others by a distance of about 15 mm. In order to prevent the coupon samples and pack materials from oxidizing because of the oxygen in the air, the retort was sealed with a lid made of alumina. Alumina cement was used to attach the lid to the retort and then dried at room temperature for about 12 hrs. After drying, the pack retort was heated in a horizontal tube furnace for the pack aluminizing process. To avoid influence of oxygen on the deposition process during aluminizing, argon gas was kept flowing into the tube furnace at 70 ml/min.

The heating cycle of the aluminizing process involved preheating at 200 °C for 1 hr to remove residual moisture in the pack. It took about 9 min to reach this temperature from room temperature. Subsequently, the temperature of the furnace was increased to 900 °C and kept at this temperature for 10 hrs for the aluminizing process to proceed. It took about 30 min to reach the aluminizing temperature from the preheating temperature. Furnace cooling was used to bring the coated samples to a temperature close to room temperature. The time for the pack retort to reach room temperature was about 11 hrs. The coated samples were removed from the pack retort and cleaned with acetone in an ultrasonic cleaner. A series of pack aluminizing processes with a similar procedure were conducted to obtain 12 coated samples, which were used for the interdiffusion test in the subsequent stage. One coated sample was selected for analysis in as coated condition.

The interdiffusion test was carried out by heating the as coated samples in a tube furnace at three different temperatures, i.e. 800 °C, 900 °C and 1000 °C, each for 5, 10 and 22 hrs. In order to avoid oxidation during the interdiffusion test, the furnace was purged with high-purity argon. After being removed from the furnace, the as coated as well as the heated samples were prepared for a metallography analysis. Each sample was put in an epoxy resin mounting so that the cross section of the samples was exposed for thickness measurement of the coating layers. The samples were polished gradually up to 0.2 micron using abrasive paper and diamond paste. After cleaning in acetone, the samples were etched in a mixture of 10% HF, 5% HNO₃ and 85% H₂O for microstructural analysis.

An optical microscope was used to observe the general microstructural condition of the coatings on the samples, as well as the thickness of the layers found in the coatings. Further, detailed microstructural observation and analysis was carried out using an energy dispersive X-ray spectroscope (EDX) attached to a scanning electron microscope (SEM). The thickness of the interdiffusion layer of each sample was measured 20 times to obtain the average.

3 Results and Discussion

The microstructure of the as coated sample and the corresponding EDX spectra of the coatings are given in Figure 2. The results of the EDX analysis gave concentration profiles of the elements across the coating and nearby substrate, as shown in Figure 3. These figures show that the as coated sample consisted of a coating identified as TiAl₃. The thickness of this layer was about 65 μ m. From a thermodynamic point of view, a layer of TiAl₂ should coexist with the TiAl₃. However, such a TiAl₂ layer could not be clearly identified in the SEM

micrograph. A very thin layer of TiAl_2 is believed to be visible underneath the TiAl_3 layer.

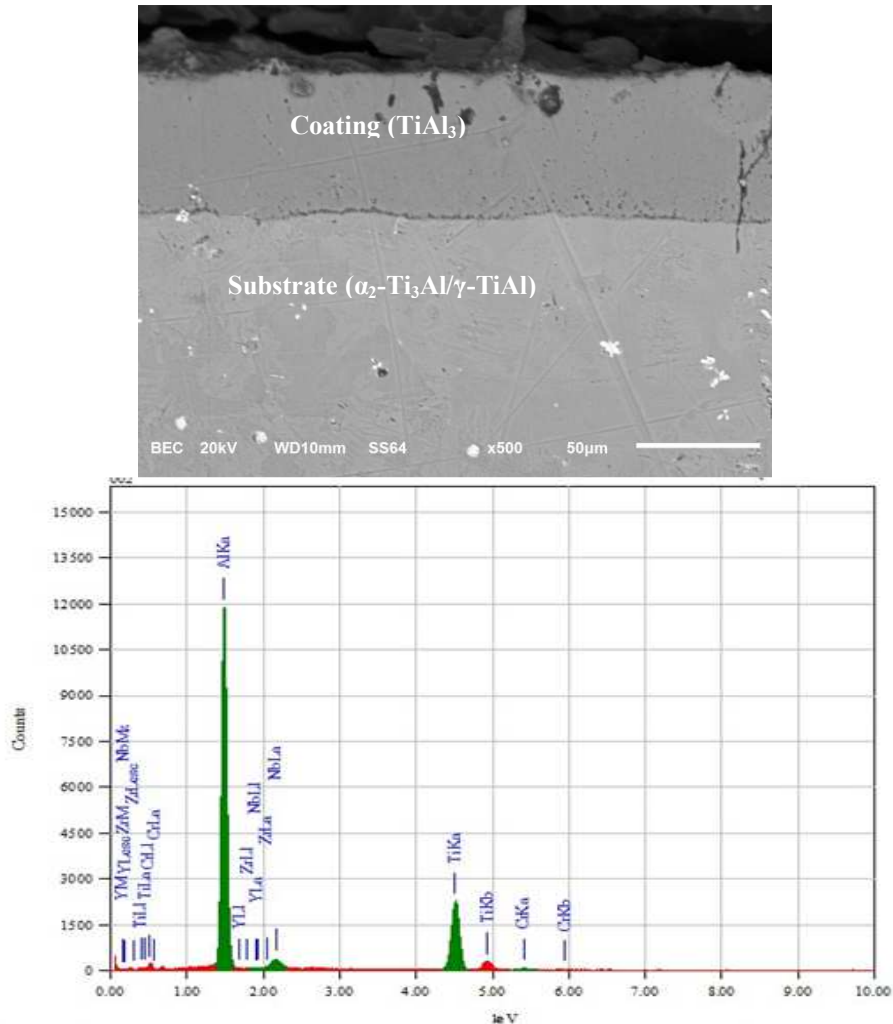


Figure 2 Microstructure and corresponding EDAX spectra of the as coated sample[12].

Heating the as coated samples at 800 °C, 900 °C and 1000 °C caused interdiffusion between the substrate of $\alpha_2\text{-Ti}_3\text{Al}/\gamma\text{-TiAl}$ and the coating of essentially TiAl_3 to proceed. The main elements in the system, i.e. Al and Ti, showed predominant elements in the interdiffusion process. Aluminium diffused from the coating to the substrate, while Ti diffused in the opposite direction as the chemical potential or concentration of aluminium in the coating

was higher than that of titanium, while the concentration of titanium was higher in the substrate compared with that in the coating, as can be seen clearly in Figure 3.

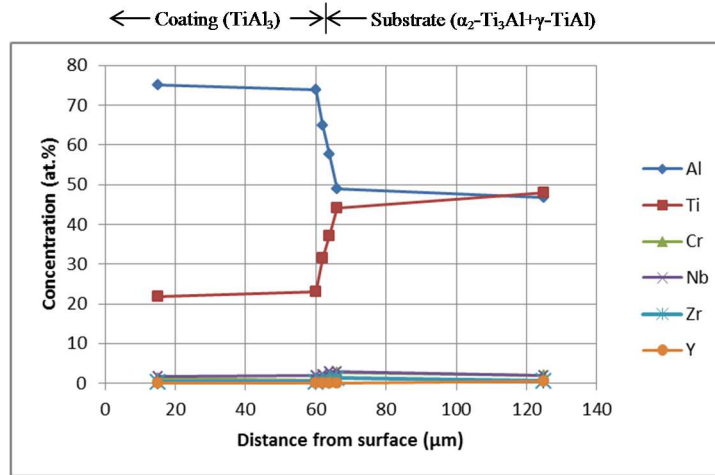


Figure 3 Concentration profiles of elements in as coated sample.

The alloying elements diffuse according to the activity of each element and the interaction behavior among the elements in the alloy system. The chemical potential of a given element, μ_i , is related to its activity by the following relationship as shown in Eq. (1),

$$\mu_i = \mu_i^0 + RT \ln a_i \quad (1)$$

where μ_i^0 represents the chemical potential of element i in its standard state, normally corresponding to 1 atm pressure and pure i (its mole fraction equals 1). This relationship shows that the higher the activity, the higher the chemical potential will be (i.e. less negative); as consequently, the higher the tendency for the element to diffuse, since by definition the intrinsic flux of element i is proportional to its potential gradient ($\frac{\partial \mu_i}{\partial x}$), mobility (M_i), and concentration (C_i), as given in the following Eq. (2)[12],

$$J_i = -M_i C_i \frac{\partial \mu_i}{\partial x} \quad (2)$$

or in the following Eq. (3):

$$J_i = -M_i C_i RT \frac{1}{a_i} \frac{da_i}{dx} \quad (3)$$

The atomic mobility of component i (M_i) is directly related to its intrinsic diffusivity (D_i). The interdiffusion between elements, especially Al and Ti, alter the microstructure of the coated system.

This interdiffusion process increases the TiAl_2 layer as the interdiffusion zone, as indicated in Figure 4, with corresponding concentration profiles for all elements involved given in Figures 5 to 7. The average thicknesses of the interdiffusion zone in the samples after heating at 800, 900 and 1000 °C for 22 hrs were 2.25, 6.82 and 21.49 μm respectively, indicating that the TiAl_2 layer grew exponentially with temperature.

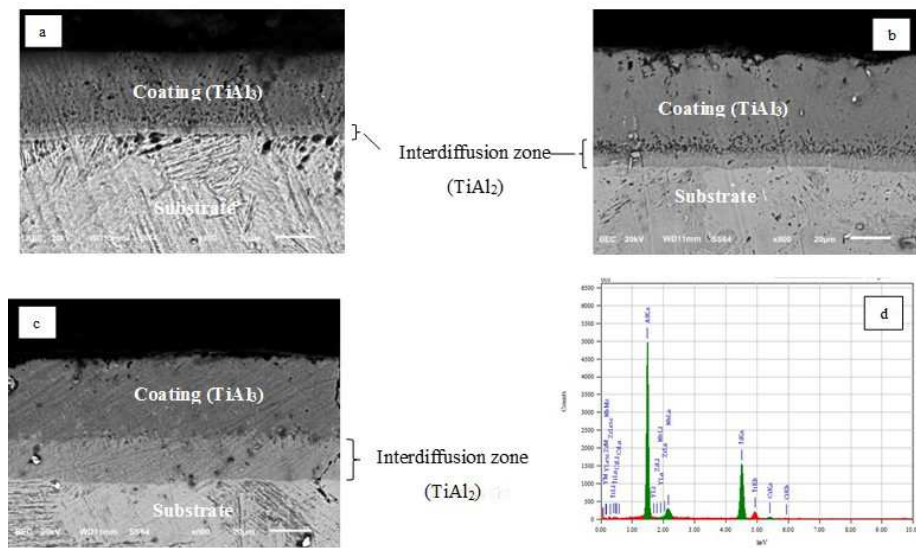


Figure 4 Microstructure of coating substrate after heating simulation for 22 hrs at different temperatures: (a) 800 °C, (b) 900 °C, (c) 1000 °C, and (d) corresponding EDAX spectra of the interdiffusion zone (TiAl_2 layer) [8].

Plots of TiAl_2 interdiffusion layer against diffusion time at these heating temperatures are given in Figure 8. The curves clearly show a parabolic relationship, which indicates diffusion has played a role in the formation of the interdiffusion layer. In this case, the rate of interdiffusion layer growth reduced with time. However, this reduction in diffusion rate depends strongly on the microstructure and temperature [12]. As this interdiffusion process occurred by vacancy diffusion mechanism due to unbalanced diffusion of Ti and Al atoms, Kirkendall voids were found in the TiAl_3 layer adjacent to the TiAl_2 layer. This confirms that the diffusion rate of Al is higher than that of Ti in TiAl_3 as previously confirmed by Van Loo and Rieck [9], i.e. the ratio between

diffusivity of Al and Ti in TiAl is approximately 3 and approaches infinity in TiAl₂ and TiAl₃.

It can be seen in Figure 4 that the enlargement of the TiAl₂ layers was followed by a recession of the TiAl₃ layer as the main phase of the coating process. Nevertheless, this study also revealed that apart from growth of the TiAl₂ interdiffusion layer at the beginning of the interdiffusion process, the TiAl₃ layer experienced enlargement before receding, as can clearly be seen from the plots of TiAl₃ thickness vs. heating time shown in Figure 9. The enlargement of the TiAl₃ thickness at the beginning of heating before it recedes is clearly shown by the samples heated at 800 and 900 °C. At 1000 °C, however, no TiAl₃ enlargement is visible. Nevertheless, it is believed that enlargement of the TiAl₃ layer in the sample heated at 1000 °C occurred after less than 5 hrs.

The concentration profiles of the elements in the samples heated for 22 hrs at different temperatures are shown in Figures 5 to 7. The formation of the interdiffusion layer of TiAl₂ between the TiAl₃ and the substrate is indicated by the concentration of the elements, typically in the range of about 56-68 at% of Al. The concentration of Al in this TiAl₂ being lower than the equilibrium concentration is believed to be due to the higher vacancies in the Al sites than in the Ti sites in the TiAl₂ crystals.

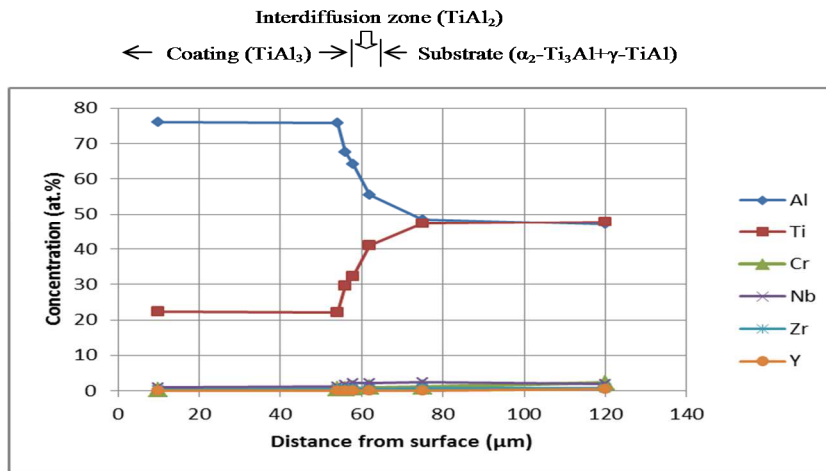


Figure 5 Concentration profiles of elements in coated sample after heating at 800 °C for 22 hrs.

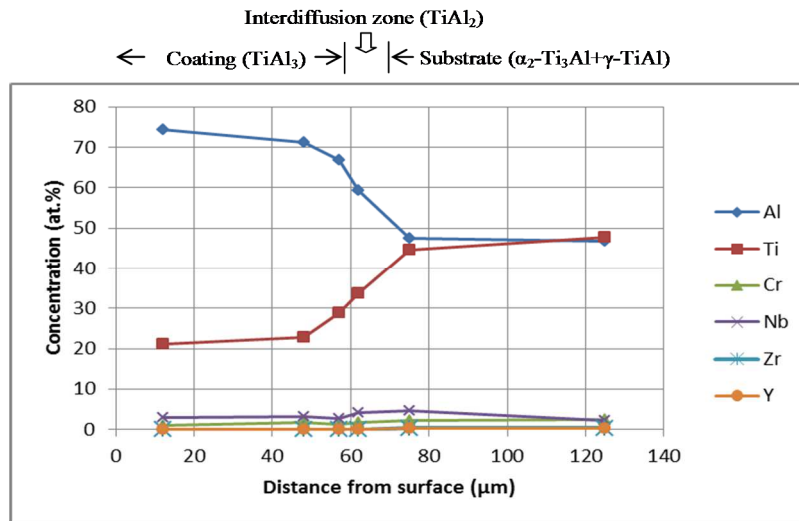


Figure 6 Concentration profiles of elements in coated sample after heating at 900 °C for 22 hrs.

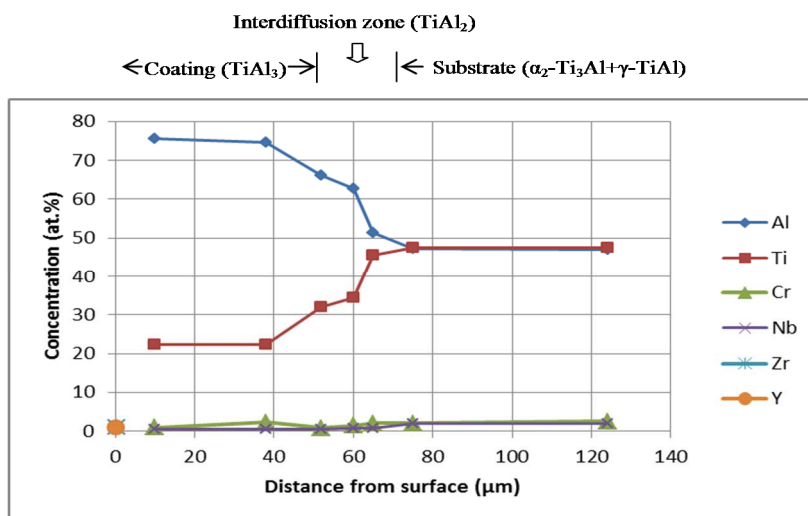


Figure 7 Concentration profiles of elements in coated sample after heating at 1000 °C for 22 hrs.

The opposite occurred for a concentration of Al higher than that of the equilibrium concentration, indicating that the vacancies in the Ti sites were

higher than in the Al. No significant concentration changes occurred for the minor elements of Cr, Nb, Zr and Y. However, even though diffusion of these minor alloying elements from the substrate into the coating is expected, these concentration profiles indicate slow diffusion of these elements during heating.

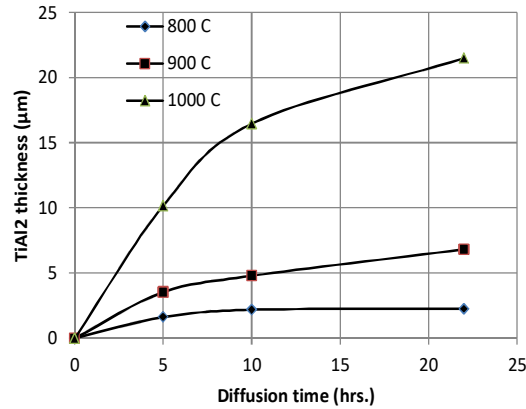


Figure 8 Kinetics of TiAl₂ growth after heating simulation at 800, 900 and 1000 °C.

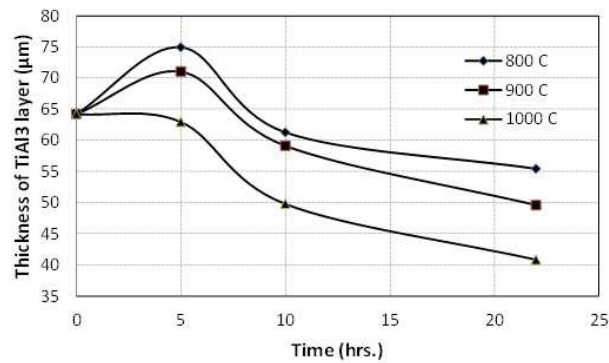


Figure 9 Kinetics of TiAl₃ growth and recession after heating simulation at 800, 900 and 1000 °C.

Figure 10 shows a diagrammatic illustration of the stage of coating degradation due to interdiffusion of elements in the coated samples during heating. Equation (3) indicates that the diffusion of component *i* is in the direction from high activity (or chemical potential) to low activity. Therefore, the relative tendencies of the elements to diffuse can be predicted from the difference in the activities of the elements in different regions, i.e. substrate and coating. When

the activity is normally directionally proportional to the concentration it is expected that aluminium diffuses from the coating to the substrate, while titanium and other alloying elements, i.e. Nb, Cr, Y and Zr, diffuse from the substrate to the coating. Figure 10(a) shows the predicted directions of the diffusion of atoms at the early stages of heating. The different concentrations of Al and Ti in the substrate and the coating indicate that Ti and Al atoms diffuse predominantly. Consequently, referring to the phase diagram of Ti-Al shown in Figure 1, at all temperatures concerned, i.e. 800, 900 and 1000 °C, a new layer of TiAl_2 should form between the coating and the substrate.

With time, as the interdiffusion proceeds, the thickness of TiAl_2 increases and this makes recession of the TiAl_3 layer the main coating phase. Meanwhile, the outward diffusion of Nb, Cr, Y and Zr atoms produces precipitates as a result of multi-element reactions. One of the important findings from this study is that aluminium-yttrium rich precipitates containing small amounts of Ti, Cr, Zr and Nb were formed in regions around the interdiffusion layer during the interdiffusion process, as shown in Figure 11. The typical chemical formula of the particle is $65\text{Al}25\text{Y}3\text{Ti}2\text{Cr}2\text{Zr}3\text{Nb}$. The binary phase diagram of Al-Y [13] indicates several intermetallic compounds of Al_xY_y type, where x and y are equal to 1 or higher. The most appropriate intermetallic compound that meets the composition of the precipitate is Al_2Y . In this case, other predicted elements mostly partition in the position of Y, but a small portion are also believed to partition in Al lattice points.

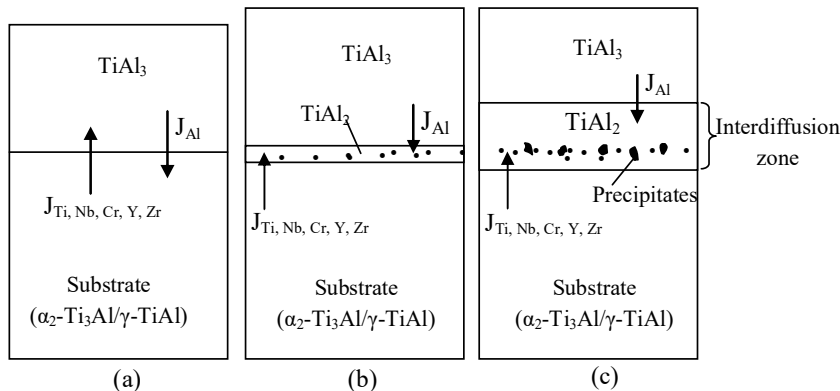


Figure 10 Schematic illustration of stages in coating degradation due to interdiffusion of Al and Ti atoms during heating: (a) beginning of interdiffusion, (b) enlargement of TiAl_3 and formation of TiAl_2 layer, and (c) recession of TiAl_3 and enlargement of TiAl_2 layer.

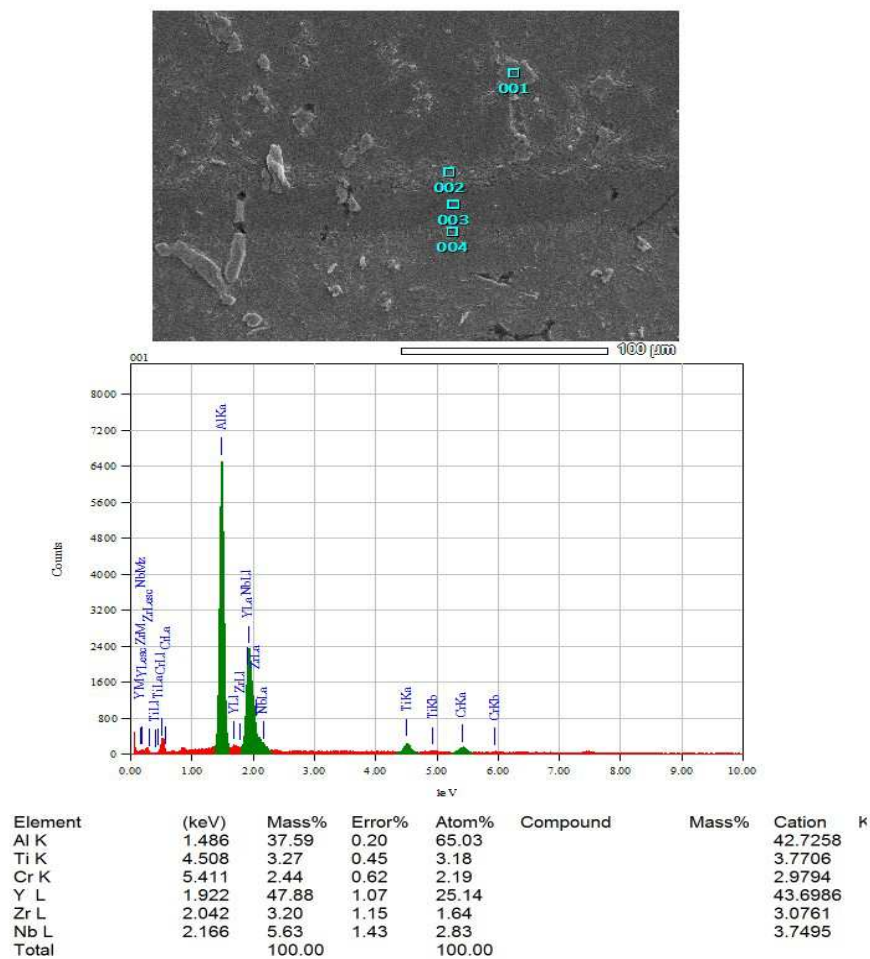


Figure 11 EDX analysis for particle precipitates at regions closed to the interdiffusion zone found in the coated sample after heating at 900 °C for 22 hrs.

Based on the work done by Wagner [14], the thickness of the TiAl₂ interdiffusion layer at a certain time, for all temperatures in this study, can be expressed by the following general equation as shown in Eq. (4) [11]:

$$\Delta x = kt^n \tag{4}$$

or alternatively, it can be written as follows in Eq. (5):

$$\ln \Delta x = n \ln t + \ln k \tag{5}$$

where Δx is the thicknesses of the TiAl_2 interdiffusion layer, k is the rate constant of the interdiffusion growth process, t is the interdiffusion time, and n is the kinetic exponent. Table 1 shows the values of Δx and t at different temperatures for the present study.

Table 1 The values of thickness of TiAl_2 layer at different time and temperatures.

Temperature (°C)	Time (hr)	Thickness of TiAl_2 layer (μm)
800	5	1.63
	10	2.19
	22	2.25
900	5	3.54
	10	4.79
	22	6.82
1000	5	10.17
	10	16.46
	22	21.49

When the values listed in Table 1 are substituted into Equation (5) and plots of $\ln \Delta x$ versus $\ln t$ for different temperatures are made, the results shown in Figure 12 are obtained. From the linear regression equations of the three graphs, the exponent n of each temperature was 0.2134, 0.4427, and 0.5012 for temperature at 800, 900, and 1000 °C respectively. It has been normally accepted that an n value of 0.5 suggests a TiAl_2 layer growth process controlled by diffusion, while a value of n higher than 0.5 indicates that the surface is predominantly controlled by TiAl_2 layer growth. The results show that heating at 1000 °C gives an n value of close to 0.5, while at 900 °C the n value is slightly smaller than 0.5, and the value of n at 800 °C is relatively far below 0.5. The kinetic exponents for binary diffusion couples of Ti-Al found by Van Loo and Rieck [9] varied between 0.25 and 0.5.

The rate constants k for different temperatures can be obtained from the graphs in Figure 12. The values of k for 1000, 900 and 800 °C were $4.76 \times 10^{-4} \mu\text{m}/\text{sec}^{0.5}$, $1.73 \times 10^{-4} \mu\text{m}/\text{sec}^{0.4}$ and $1.22 \times 10^{-4} \mu\text{m}/\text{sec}^{0.2}$, respectively. Other studies on interdiffusion layer growth on TiAl systems were conducted in diffusion couples and less complex systems of binary TiAl [9,10,11]. The values of k for TiAl_2 growth in a binary TiAl system at 700, 800 and 900 °C obtained by Chu and Wu [10] were $1.63 \times 10^{-4} \mu\text{m}/\text{sec}^{0.5}$, $3.19 \times 10^{-4} \mu\text{m}/\text{sec}^{0.5}$ and $9.3 \times 10^{-4} \mu\text{m}/\text{sec}^{0.5}$, respectively. Normally, they fit for $n = 0.5$, which represents purely diffusion-controlled growth. The lower value of k for heating at 1000 °C of this study compared with that obtained by Chu and Wu for heating at 900 °C indicates that the growth of the interdiffusion layer in a more complex multi-

component coated system is slower compared to that of a simpler binary system. The elements involved and the microstructure constituents occurring in this more complex alloy system are believed to have played a role in decreasing the rate of interdiffusion.

The kinetic exponents of n found in the present study decreased with lowering temperatures showing that reduction in the growth rate of TiAl₂ as interdiffusion layer decreased with increasing temperatures. It is believed that alloying elements Cr, Nb, Zr and Y played a significant role in the reduction of the interdiffusion process rate at lower temperatures. At higher temperatures, however, the effect of the alloying elements in reducing the interdiffusion process rate became less significant due to the increasing of the distance between atoms in the crystals of the TiAl₂ with temperature. Therefore, apart from ineffective diffusion along high-diffusion paths such as grain boundaries and the interface between matrix and precipitate particles, the blockage of precipitates particles for the movement of Ti and Al during interdiffusion is less sensitive at high temperatures.

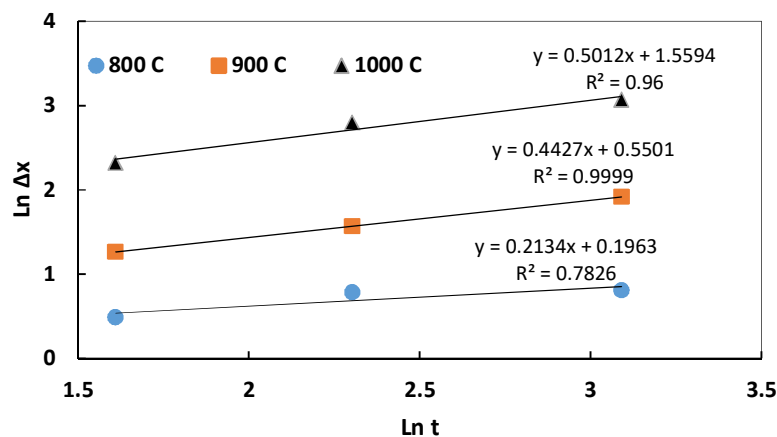


Figure 12 Growth kinetics of interdiffusion TiAl₂ layer at 800, 900 and 1000 °C.

4 Conclusion

The investigation of the effects of heating aluminide coated α_2 -Ti₃Al/ γ -TiAl alloys with a composition of Ti-47Al-2Nb-2Cr-0.5Y-0.5Zr indicated that a unique behavior of interdiffusion occurred during heating of TiAl₃ pack aluminide coatings on a substrate of α_2 -Ti₃Al/ γ -TiAl. Acting as the main layer for providing protection during heating of titanium aluminide coatings, the

TiAl₃ experienced homogenization that enlarged its thickness. However, with the completion of the homogenization stage, the TiAl₃ layer entered into a stage of recession due to reduction of its thickness and the growth of TiAl₂ phase as an interdiffusion layer between the substrate and the TiAl₃. The exponent n of interdiffusion layer growth at 800, 900, and 1000 °C was 0.2134, 0.4427, and 0.5012, while the rate constant k was $4.76 \times 10^{-4} \mu\text{m}/\text{sec}^{0.5}$, $1.73 \times 10^{-4} \mu\text{m}/\text{sec}^{0.4}$ and $1.22 \times 10^{-4} \mu\text{m}/\text{sec}^{0.2}$, respectively. Addition of alloying elements at 2% Nb, 2% Cr, 0.5% Zr and 0.5 %Y to the substrate reduced the interdiffusion rate only significantly at lower temperatures, such as 800 °C. The interaction of these alloying elements and interdiffusion of Ti and Al base metals produced particles precipitated adjacent to the interdiffusion zone of the type Al₂Y with a chemical composition of about 65Al25Y3Ti2Cr2Zr3Nb.

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